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Cyclic aging analysis of CFRP and GFRP composite laminates

Mostafa Moazzami[1], MR Ayatollahi[1], Alireza Akhavan-Safar[2], Sofia Teixeira de Freitas[3] and Lucas FM da Silva[4]

Abstract

When a composite laminate is subjected to humidity, moisture diffusion occurs depending on the number and thickness of the lamina. Water diffusion changes the mechanical response of laminates and usually causes a significant reduction of the mechanical properties of the composite specimens in ocean structures. One of the most important mechanical properties of laminates is flexural stiffness which should be considered in the design procedure. Despite the extensive research on single cycle aging of composites, cyclic aging of these materials is less explored. The aim of the current research is to investigate the variation of mechanical properties of carbon fiber reinforced polymer (CFRP) and glass fiber reinforced polymer (GFRP) composites as a substrate in adhesive joints with the same initial flexural stiffness values subjected to cyclic wet/dry aging conditions for long-term structural applications. The matrix used in the CFRP and GFRP composites are based on epoxy and vinyl ester, respectively. Both unaged and cyclically aged samples were characterized by tensile and three-point bending tests. In order to simulate the moisture absorption condition of composites in adhesive joints, one side of the composite laminates was sealed with aluminum foils and three sides were exposed to humidity. The interaction between the composite thickness and the number of aging cycles was also investigated. The experimental results show that in cyclic aging condition, the reduction of flexural stiffness in CFRP is more than GFRP laminates and GFRP laminates is more suitable for ocean applications.

Keywords

Composite laminate, cyclic aging, water diffusion, tensile and flexural properties

Introduction

Composite laminates used in different structures often experience humid conditions in service which can change their properties. Some examples are marine, offshore, and aircraft applications. For marine environments, composites have an interesting potential, due to their favorite mechanical properties such as high strength, good fatigue durability, and design ability.[1] Additionally, these materials are increasingly used in marine equipment[2,3] and navy ships[4,5] due to their light weight and good corrosion resistance. In these applications, composite laminates are exposed to environments with different temperatures and humidity levels. In wet conditions, composite laminates with a polymer matrix (such as epoxy, polyurethane and vinyl ester) uptake moisture by a diffusion process, which can drastically affect the mechanical and chemical properties of these materials.[6–12] The fluctuation of temperature and moisture absorption can create dimensional variation in laminated composites with epoxy matrix known as hygrothermal effects.[13] Accordingly, the analysis of the aging of composite materials is of great importance.

Moisture diffuses by two mechanisms that consist of direct diffusion of moisture into the resin through micro-cracks or voids, and capillarity diffusion at the fiber/matrix

[1]Fatigue and Fracture Research Laboratory, Center of Excellence in Experimental Solid Mechanics and Dynamics, School of Mechanical Engineering, Iran University of Science and Technology, Tehran, Iran
[2]Institute of Science and Innovation in Mechanical and Industrial Engineering(INEGI), Porto, Portugal
[3]Structural Integrity & Composites Group, Faculty Aerospace Engineering, Delft University of Technology, Delft, The Netherlands
[4]Departamento de Engenharia Mecanica, Faculdade de Engenharia da Universidade do Porto, Porto, Portugal

Corresponding author:
MR Ayatollahi, Mechanical Engineering, Iran University of Science and Technology, Narmak, Tehran 16844, Iran.
Email: m.ayat@iust.ac.ir

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(glass/epoxy) interface.[14] Accordingly, two typical mechanisms have been proposed to describe the effects of moisture diffusion in composite structures: (a) degradation of the fiber/matrix (carbon/phenolic) interface[15] and (b) matrix plasticization.

As mentioned above, moisture absorbed by the matrix of composites can create a plasticization effect and reduce the glass transition temperature (T_g) of the epoxy resin.[16–19] The presence of moisture in composite materials can lead to reversible or irreversible effects. Moisture can cause dimensional variations or residual stresses due to dimensional mismatch. Experimental studies have demonstrated that moisture diffusion can significantly impact the fiber/matrix (glass/epoxy) interface.[20–23] Epoxy resin has a higher tendency for moisture uptake, leading to volume expansion, whereas fibers do not absorb moisture.[16] This difference in moisture absorption between the resin and fibers can result in resin swelling and cause stress and fiber/matrix (carbon/epoxy) interface debonding.[24] Additionally, moisture can disrupt the matrix/fiber (glass/epoxy) bonding at the interface and even change the chemical structure of the bonded region, thus affecting the composite's overall performance.[2,25–28] Previous studies have shown that some of these moisture-induced mechanisms are reversible, such as plasticization,[29] swelling,[13] and reduction of glass transition temperature.[30] However, some mechanisms are irreversible, including micro-cracks in the matrix or even fiber/matrix interface debonding.[16] Therefore, it is crucial to consider the effects of moisture on the fiber/matrix interface and the overall performance of composite materials.

Different factors such as fiber volume fraction, temperature, fiber orientation, exposed area, diffusivity, and surface protection can affect the water absorption of composites with polyester and vinyl ester matrix.[33,32,31] Rao et al.[34] believe that in a quasi-isotropic composite laminate with epoxy matrix, the moisture and temperature have almost the same effects on the properties of the composite in transverse, shear, and longitudinal directions.

Compared to monotonous moisture diffusion, studies on cyclic aging of composite components is relatively rare. In the case of the effect of cyclic moisture of the matrix, experimental results show that the molecular structure of the resin chain in epoxy matrix composites can change with cyclic aging.[36,35] These variations lead to some changes in moisture diffusion mechanisms in the epoxy matrix in cyclic moisture absorption conditions.[37] The variation of moisture diffusion leads to complex moisture absorption and desorption mechanisms. Sun et al.[26] investigated the effects of temperature variation and cyclic moisture uptake on the shear strength of carbon fiber/bismaleimide composites. They concluded that absorption/desorption cycles lead to an increase in the diffusion coefficients and decreases the saturated concentration. Lin and Chen[38] developed a molecular dynamics approach and an experimental method to study the processes of monotonous and cyclic aging for an epoxy. The experimental results showed that the reabsorption speed is faster than the initial absorption. They also found that the process of cyclic moisture diffusion does not exactly fit the Fick's law. Yagoubi et al.[39] investigated the cyclic moisture diffusion process for a thermosetting epoxy resin. They proposed a phenomenological reaction–diffusion model to describe this cyclic diffusion process. Cyclic moisture aging of composite materials with an epoxy matrix exhibits a quite complex behavior and has not been well understood and predicted by models. The main reason is that during the moisture uptake of composite laminates, chemical changes like chemical scission and hydrolysis, as well as physical changes like swelling and micro-cracks propagation occur. Some of these phenomena are reversible and some others are irreversible and can degrade the properties of composite materials with an epoxy matrix after drying.[40,37] Cyclic aging can induce stresses and create micro-cracks within the polyester matrix, which causes even greater water absorption in the following cycles.[41] In previous investigations, authors have mainly focused on the monotonous or non-cyclic moisture absorption mechanisms in composites with epoxy and vinyl ester matrix.[43,42,13] However, in real applications such as the marine sector, the aging process is usually cyclic and often one side is isolated such as adhesively bonded composites in retrofitting of marine structures. Previous researchers have focused on the variation of the mechanical properties under two-sided cyclic aging conditions with the maximum of five aging cycles.[37,29] Further investigation is needed to understand the mechanical behavior of composite laminates under one-sided cyclic moisture absorption with a high number of aging cycles. The current literature indicates a lack of sufficient evidence regarding the changes in tensile and flexural properties of composite laminates subjected to one-sided isolated aging conditions.

The aim of the current research is to experimentally analyze the mechanical properties of CFRP and GFRP composites specimens subjected to different cyclic aging conditions. The design parameters of composite structures, depend on loading conditions and the mechanical properties of the structures should be controlled based on the operating loads. There are many composite structures in which the dominant forces are induced by bending stresses such as adhesive joints with composite substrates. In these structures, one of the most important mechanical properties showing capacity of bending load bearing is flexural stiffness, which should be controlled in different aging conditions during the structure service time. This parameter considers flexural modulus and laminate thickness simultaneously, and should satisfy some criteria to show good performance in the structure. In order to obtain a certain flexural stiffness, based on the properties of laminate, the thickness can be calculated. Because of the importance of flexural stiffness property of composites laminates in designing process, the CFRP and GFRP specimen with the same initial flexural stiffness was studied. To achieve this, at the first step, some CFRP specimens with three different thicknesses (8, 16 and 24 layers) were manufactured and exposed to 7-day wet/7-day dry aging cycles. In this step, the degradation of mechanical properties of CFRP specimens as a function of the thicknesses was investigated. Then in the next step, CFRP specimens with 24 layers and GFRP laminates were exposed to 14-day wet/7-day dry cyclic ageing. The thickness of the GFRP specimens was calculated in order to have the same flexural stiffness as the CFRP samples with 24 layer. For all specimens the unaged and cyclic aged samples were characterized by tensile and three-point bending tests and the mechanical properties were obtained for different aging and geometrical conditions. It should be mentioned that the moisture diffusion mechanism in composite laminates is strongly dependent on the used matrix which are epoxy and vinyl ester in this investigation.

Experimental details

Materials and geometries

In this research two categories of composite laminates were fabricated and tested. For CFRP specimens, unidirectional T300 carbon fiber-reinforced composites (UD-CFRP) with three different thicknesses (8, 16 and 24 layers) with stacking sequence of $[0]_8$, $[0]_{16}$ and $[0]_{24}$ were made. The average thicknesses of these three specimens are 1.73, 3.43, and 5.48 mm, respectively. The CFRP composites were manufactured using Araldite® LY 5052/Aradur® 5052 supplied by Huntsman. Resin and hardener were mixed with a weight ratio of 100/38 (resin/hardener) using a centrifuge mixing machine. All the specimens were cured according to the datasheet of the considered epoxy, 1 day at 23°C followed by 1 h at 100°C in an oven. The GFRP specimens were fabricated with quadraxial E-glass fabric consisting of a stacking of four unidirectional (UD) layers of E-glass lamina with the orientations −45°/90°/+45°/0°. GFRP composites were made by vacuum infusing the fabric stacking sequence with DION® 9500 rubber modified epoxy-based vinyl ester resin supplied by Reichhold. The laminate was cured for 1 day at 23°C followed by 12 h at 60°C in an oven. The research first investigated the mechanical properties of CFRP composites with varying thicknesses during cyclic aging. Then, the bending parameters of 24-layer CFRP and GFRP composites were compared before and after cyclic aging. Since flexural stiffness is an important parameter, the thickness of the GFRP composite was calculated to achieve the same initial flexural stiffness as the CFRP. It's important to note that the behavior of composite laminates under bending conditions depends on both the flexural modulus and the thickness of the laminates. As a result, flexural stiffness of composite components is a crucial parameter for analysis of structures including bonded composite laminates during the service time in different aging condition. Accordingly, in this section, the CFRP and GFRP with the same initial flexural stiffness were investigated. The experimental results show that, the flexural stiffness of which composite laminate with the same initial flexural stiffness reduces more significantly. Therefore, the thickness of the GFRP specimens was calculated based on the flexural stiffness of the CFRPs with 24 layers. Accordingly, the thickness of the GFRP composites was obtained using the following relation:[44]

$$\left(E_0^f h^3\right)_{CFRP} = \left(E_0^f h^3\right)_{GFRP} \tag{1}$$

where, h and E_0^f are the specimen thickness and the flexural modulus, respectively. Based on the literature, flexural modulus of unaged CFRP (UD-CFRP with 24 layers thickness $[0]_{24}$) and unaged GFRP (quadraxial E-glass fabric consisting of a stacking of four unidirectional (UD) layers of E-glass lamina with 10 layer thickness, $[45/90/+45/0]_5/[45/90/+45/0]_5$) has been obtained as 81,000 MPa[45] and 21,000 MPa,[44] respectively. Considering equation (1), the thickness of the GFRP specimens must be 8.6 mm. This thickness can be obtained by stacking 10 quadraxial layers. After curing, the manufactured CFRP and GFRP plates were cut into 5 mm × 120 mm specimens. The considered specimens are smaller than the standard specimens in order to accelerate the effect of aging on the mechanical properties of the test samples. The geometry of the specimens for different thicknesses are shown in Figure 1.

The manufactured specimens were exposed to cyclic aging after sealing one side of the specimens. The cyclic aging procedure is explained in the next section. In order to minimize experimental errors, for the specimens under tensile and bending tests, five and three samples were manufactured and tested in each aging condition.

Cyclic aging procedure

Two cyclic aging processes were considered. In The first process each cycle consists of 7 days ageing followed by 7 days drying. This aging cycle was repeated twelves times. Consequently the total duration of the aging process is 14 × 12 = 168 days as schematically shown in Figure 2.

In order to investigate the effect of laminate thickness on the variation of flexural parameters, CFRP specimens with different thicknesses were tested. In this aging process, CFRP specimens were immersed in distilled water at a constant temperature of 50 ± 2°C for 7 days and then were exposed to a dry environment at a constant temperature of 50 ± 2°C for 7 days at a relative humidity of 4% using a box containing silica gel powder. Each consecutive immersion-drying

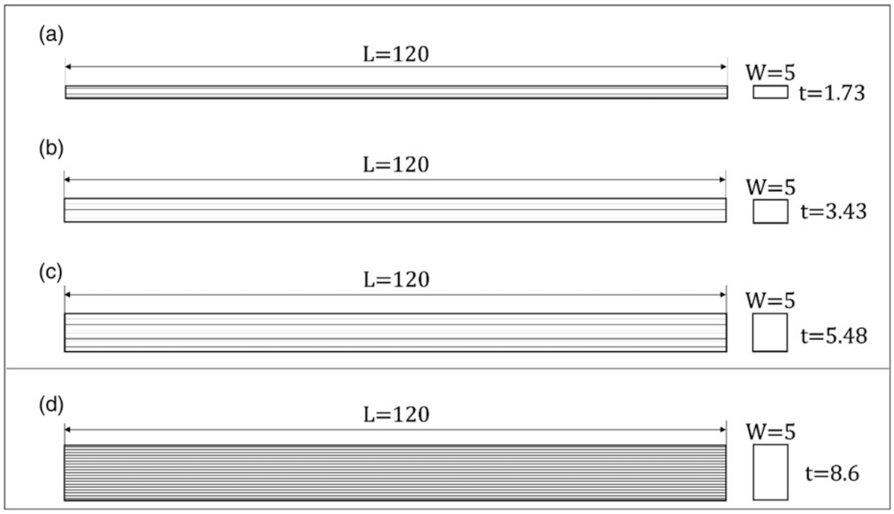


Figure 1. Tested CFRP specimens with (a) 8 ($[0]_8$), (b) 16 ($[0]_{16}$) and (c) 24 ($[0]_{24}$) layers, and (d) GFRP specimen with 10 layers ($[0° +45° 90° -45°]_5/[+45° 90° -45° 0°]_5$) (dimensions in mm).

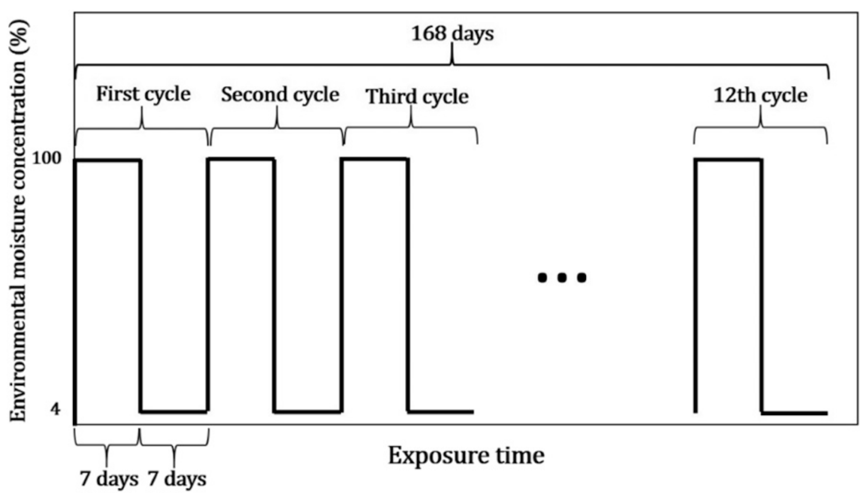


Figure 2. Schematic of the 7-day wetting/7-day drying cyclic moisture conditioning process.

represents one cycle and this procedure was repeated 12 times (12 cycles).

The second cyclic aging procedure includes 14 days wetting followed by 7 days drying. This cyclic aging was applied on CFRP and GFRP specimens with the same initial flexural stiffness in order to compare the flexural stiffness degradation during the aging process. The specimens were exposed during 14 days by immersing in distilled water, followed by 7 days drying at a relative humidity of 4% for 7 days using a box containing silica gel powder for each cycle, and for a total aging time of 84 days, as shown in Figure 3. The aging and drying temperatures were both set to 25 ± 1°C. Each consecutive wetting-drying shows one cycle and this wetting-drying process was repeated 4 times (4 cycles).

In order to investigate the variation of the mechanical properties of the manufactured composites under cyclic moisture aging, one side of the specimens was sealed with aluminum foil and adhesive before aging. In this condition, moisture uptake occurs through three sides of the specimen. In reality, there is some moisture diffusion that occurs on the fourth side via the adhesive layer between the composite and aluminum foil as well as the interface regions. However, this small amount of moisture diffusion can be disregarded when compared to the diffusion that occurs on the other three sides. Three repeats of each CFRP and GFRP substrates were tested in tensile and three-point bending loading conditions at different aging levels. The details of the experimental tests are described in the next sections.

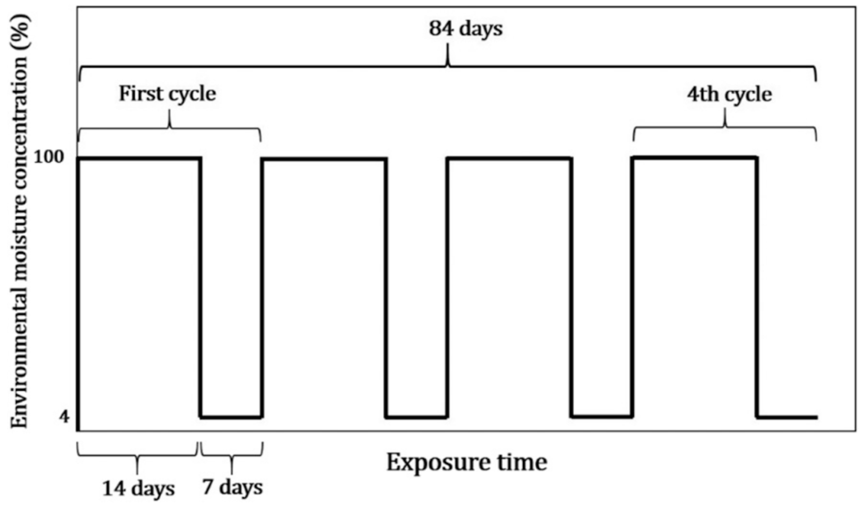


Figure 3. Schematic of the 14-day wetting/7-day drying cyclic aging process.

Tensile tests

The specimens were tested before aging and after different numbers of cyclic aging using a universal tensile testing machine (Santam Inc., Iran) and the amount of load was calculated by a 10 kN load cell. All of the tests were performed under constant displacement rate of 1 mm/min. The testing temperature was 21°C. In this process, the specimens were taken out of the water at different aging cycles, then two end tabs were bonded at each end of the specimens as shown in Figure 4.

After preparation of the specimens, they were tested using a universal testing machine. To precisely measure the strain of the samples during the tensile test, the digital image correlation technique (DIC) was used. This approach has been used extensively by previous researchers in order to calculate displacement fields.[46] During the tensile tests, using the DIC the strain was calculated. The DIC setup includes a tensile testing machine, photo-capturing equipment, and an image processing software. A high resolution digital camera and a Macro USM fixed lens was employed to capture the images during the test. The digital camera was placed at a distance of 50 cm perpendicular to the specimen. Two light sources were used to elucidate the surface of the specimens, as shown in Figure 5. It should be mentioned that before the tensile tests, specimens were dried and the aluminum foil was removed from the composite specimens.

Three-point bending test

In this study, the variation of flexural modulus and flexural strength of CFRP and GFRP composites exposed to cyclic moisture absorption and desorption were investigated using three-point bending tests. The experiments were conducted in a tensile testing machine (Santam Co., Iran) and based on ASTM D7264. All of the tests were performed under constant displacement rate of 1 mm/min and the load was recorded using 10 kN load cell. The testing temperature was 21°C Deflections of the specimens during the tests was measured using the DIC technique. The flexural strength was calculated by the following formula:

$$\sigma_f = \frac{3Pl}{2bh^2} \tag{2}$$

where σ_f is the stress at the outer layer at mid-span location, P is the force, b is the width of the specimen, l is the support span, and h is the specimen thickness (see Figure 6).

The maximum positive strain is located at the bottom surface of the middle point of the specimen and is obtained as follows:

$$\epsilon = \frac{6wh}{l^2} \tag{3}$$

where w is the maximum deflection of the specimen and ϵ is the flexural strain.

The Chord approach, which is recommended in ASTM D7264, was used to calculate the flexural modulus of the CFRP laminates. For estimation of the flexural Chord modulus, the proposed strain range is 0.002 (starting at 0.001 and ending at 0.003). The flexural modulus can be expressed as:

$$E = \frac{\Delta\sigma}{\Delta\epsilon} \tag{4}$$

As mentioned before, after drying the selected specimens, the aluminum foil was removed from the sealed side of the samples before the three-point bending tests. As the sealed surface is usually in contact with the other part of the structure or in contact with the adhesive in adhesive joints, the performance of this part is more critical. Accordingly, in all the bending tests the sealed surfaces were positioned at the bottom that experience tensile stresses.

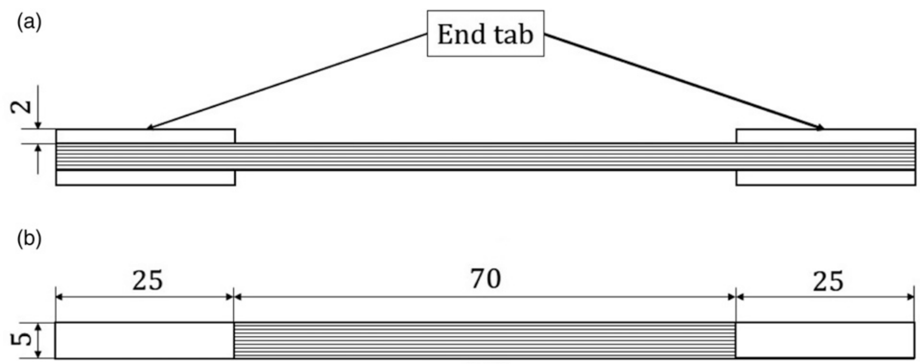


Figure 4. Geometry of tensile specimens, (a) side view, (b) top view, (dimensions in mm).

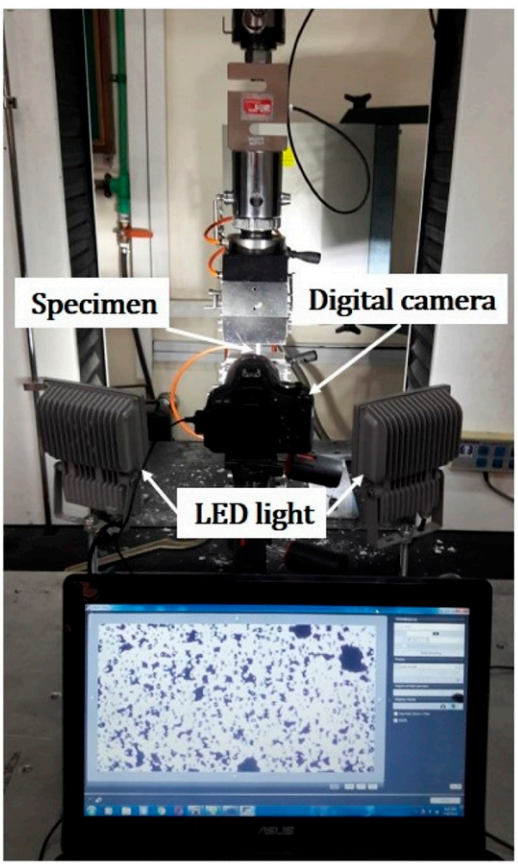


Figure 5. Outline of the tensile test setup.

Results

Elastic modulus

In this section the results obtained from the different cyclic aging procedure is presented separately.

- 7-day wetting/7-day drying process

The variation of the elastic modulus of CFRP specimens with three different thicknesses which were exposed to 12 cycles of 7-day wetting/7-day drying process obtained out of the tensile tests is shown in Table 1.

The comparison of initial elastic modulus of CFRP with different thicknesses show that, the elastic modulus of 24-layer CFRP reduces by 9% compared to the 8-layer CFRP. Most of composite fabrication equipment are designed for thin laminate fabrication process. While thick composites find applications in the marine industry, most composite fabrication components are designed to use thin composite laminates fabrication. Accordingly, the thickness of 24-layer CFRP is more than the thickness considered in most of the applications leading to some errors in the fabrication process. An example of the manufacturing issues for 24-layer CFRP is the vacuum pumps that may not function properly, resulting in voids and internal defects within the resin. These fabrication errors can cause a reduction in the elastic and flexural moduli of the CFRP as the laminate thickness increases. As it can be concluded from Table 1, a smooth negligible reduction in tensile modulus of the tested samples is observed by increasing the number of aging cycles. The observed decrease in elastic modulus is attributed to changes in the mechanical properties of the resin matrix. Moisture diffusion induces a reduction in the elastic modulus and tensile strength of the polymer matrix. However, in unidirectional laminates, where fibers bear the majority of the loads, the resulting stress distribution renders the reduction in matrix elastic modulus inconsequential to the overall elastic modulus of the composite. It shows that the cyclic aging doesn't influence the elastic modulus of the tested composites significantly. As shown in Table 1, the elastic modulus of CFRP specimens with different thicknesses are slightly different which can be due to fabrication errors. In order to show the variation of the elastic modulus in different aging conditions, the normalized elastic modulus (the

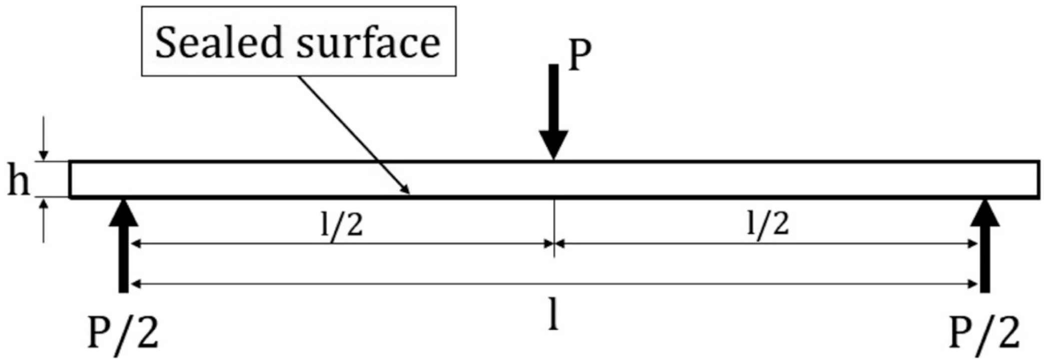


Figure 6. Schematic of three-point bending test of CFRP specimens.

Table 1. Elastic modulus for different CFRP specimen thicknesses in unaged and aged conditions.

Specimen thickness	Aging conditions	Elastic modulus (GPa)
8 layers	Unaged	85 (±2)
	3 cycles	83 (±3)
	6 cycles	81 (±5)
	9 cycles	81 (±4)
	12 cycles	82 (±2)
16 layers	Unaged	81 (±1)
	3 cycles	81 (±4)
	6 cycles	80 (±7)
	9 cycles	79 (±5)
	12 cycles	77 (±6)
24 layers	Unaged	77 (±2)
	3 cycles	76 (±4)
	6 cycles	78 (±3)
	9 cycles	75 (±6)
	12 cycles	75 (±5)

elastic modulus for aged conditions normalized by the unaged value) is presented in Figure 7.

According to Figure 7, the normalized elastic modulus in different aging cycles is close to one. The normalized elastic modulus for the 8 and 16-layer specimens are between 1 and 0.95. This parameter for the 24-layer specimen is between 1.01 and 0.97.

-14-day wetting/7-day drying process

The elastic modulus of unaged and aged CFRP and GFRP specimens after 1, 2 and 4 cycles of 14-day wetting/7-day drying obtained out of the tensile tests are reported in Table 2.

The results obtained from unaged and aged GFRP and CFRP specimens show that the elastic modulus of these specimens decreases by ageing cycles. However, the reduction in the elastic modulus is negligible and the elastic modulus can be considered constant during the cyclic aging process. As can be seen in Tables 1 and 2, the elastic modulus of CFRP and GFRP specimens for different numbers of aging cycles in one-side isolated absorption and desorption condition is almost constant and close to the unaged conditions for the tested samples. The main reason for this small variation is because of the aging process condition which is three sided aging for short cycle periods.

Flexural modulus

In this section, the flexural modulus variation of CFRP and GFRP specimens exposed to different cyclic aging procedures was investigated. Therefore, using the DIC technique, the deflection of the specimens was calculated during the tests, and then using equation (3), the flexural deformation was obtained for different load levels. As in the previous section, the results of two cyclic aging processes are presented separately.

- 7-day wetting/7-day drying process

Figure 8 shows the flexural stress–strain curves for CFRP specimens with different thicknesses exposed to 7-day wetting/7-day drying cyclic aging process.

As shown in Figure 8, in CFRP specimens for all the three thicknesses, the cyclic aging affects the flexural stress–strain curves significantly. Based on Figure 8, it is shown that the slope of the curves decreases with the number of aging cycles. A comparison of the flexural stress–strain curves shows that the variation of the flexural modulus for the 8-layer composites is less than the variation of the flexural modulus for the 16- and 24-layer composite laminates. In CFRP laminates with different thicknesses, the area of the lateral faces is different. The variation of the lateral face area affects the diffusion rate which can be considered as a main reason for the different obtained results for samples with different thicknesses. The variation of the flexural modulus for different conditions is given in Table 3.

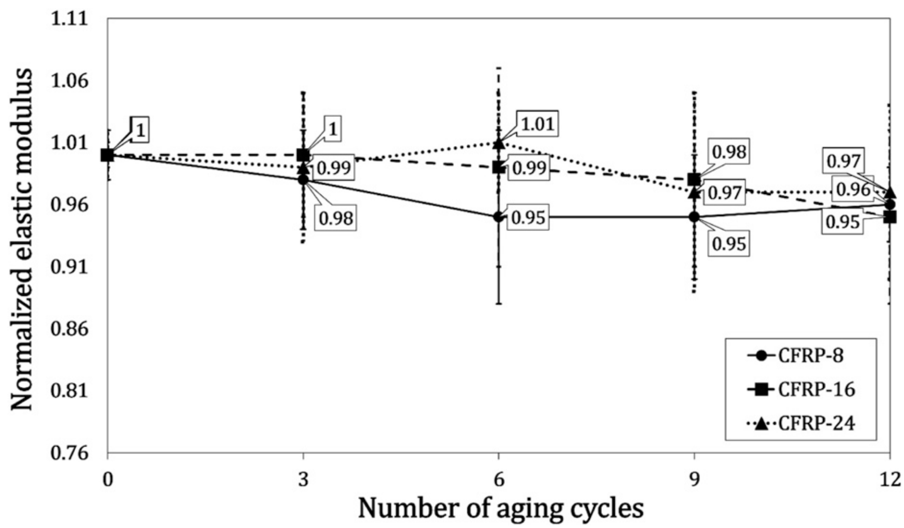


Figure 7. Normalized elastic modulus for different aging cycles and for different CFRP specimen thickness.

Table 2. Elastic modulus for CFRP and GFRP specimens in unaged and aged conditions.

Specimen	Aging conditions	Elastic modulus (GPa)
GFRP	Unaged	22 (±1)
	1 cycle	23 (±3)
	2 cycles	21 (±2)
	4 cycles	20 (±4)
CFRP	Unaged	77 (±2)
	1 cycle	72 (±4)
	2 cycles	75 (±6)
	4 cycles	72 (±5)

As can be concluded from Table 3, in the CFRP specimens the amount of flexural modulus in the 24-layer specimens is lower than 8- and 16-layer specimens. This difference can be due to fabrication errors and shear stress effects, which increase when the specimen thickness increases. In order to compare the variation of the flexural modulus for different number of aging cycles for CFRP specimens with different thicknesses, the normalized flexural modulus for each specimen is illustrated in Figure 9.

As can be found from Figure 9, in CFRP specimens the flexural modulus decreases with an increase in the number of aging cycles. However, the variation in flexural modulus is different for different specimens. Based on Figure 9, the flexural modulus in the 8-layer samples is less sensitive to the applied cyclic aging comparing with the 16- and 24-layer CFRPs. Based on the obtained results, it was found that in contrast with the tensile test results, cyclic aging can significantly reduce the flexural modulus of the composite materials up to the 60% of the unaged conditions for the tested samples. It is also concluded that the reduction in flexural modulus is almost linear for the tested CFRPs. It means that a linear relation can be assumed between the number of aging cycle and the amount of reduction in the flexural modulus of the composite materials. From Figure 9 it can be concluded that in the tested CFRPs, the influence of aging cycles on the flexural modulus is more significant for the specimens with more layers. Comparing the elastic and flexural modulus of CFRP specimens with varying thicknesses under the same boundary and aging conditions reveals a significant difference in their flexural behavior compared to their elastic modulus when subjected to different loading conditions. For example, the maximum reduction in flexural modulus and elastic modulus after 12 cycles was 23% and 5%, respectively (refer to Figures 7 and 9). Previous researchers[47] have reported this disparity in the tensile and flexural behaviors of aged composites. The mechanical behavior of laminates is affected by the stress distribution of the resin in aged composites. As stated in the Introduction, moisture absorption induces plasticization effects in epoxy resins, reducing the tensile modulus and strength of the matrix. When unidirectional composites are subjected to tensile loads along the fiber direction, the fibers bear most of the load. However, during bending tests, the aged matrix experiences tensile, compression, and shear stresses. Moreover, the strength of the matrix is reduced due to moisture diffusion. Consequently, the aged matrix can withstand a smaller bending load. Interfacial debonding is another phenomenon that can accelerate composite failure during bending tests. This type of debonding may occur during moisture adsorption due to residual stress resulting from dimensional mismatch or during bending tests due to weak interface bonding caused by capillarity diffusion at the fiber/matrix interface.

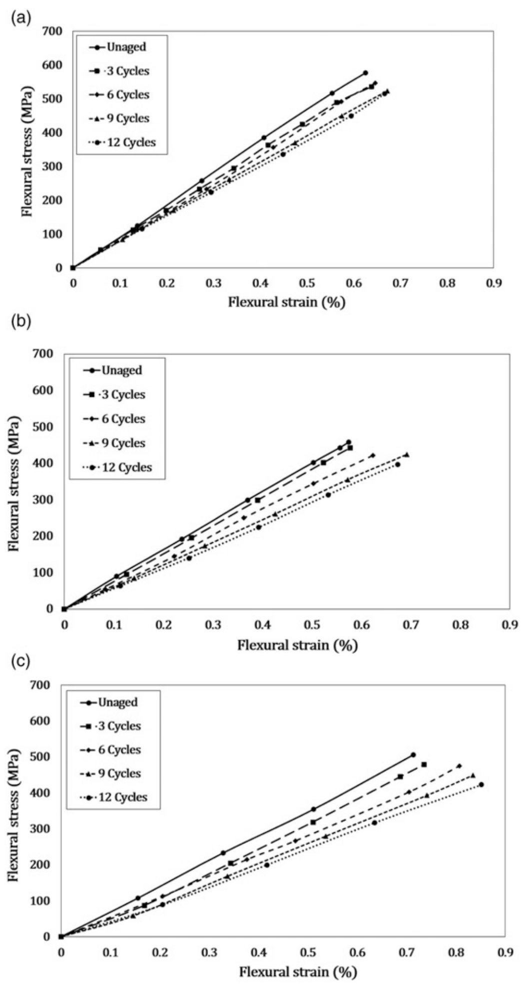


Figure 8. Variation of flexural stress–strain curves for different number of aging cycles and for CFRP specimens with (a) 8 (b) 16 and (c) 24 layers.

Table 3. Flexural modulus for different specimen thicknesses in unaged and aged CFRP specimens.

Specimen thickness	Aging conditions	Flexural modulus (GPa)
8 layers	Unaged	90 (±5)
	3 cycles	89 (±3)
	6 cycles	83 (±4)
	9 cycles	79 (±3)
	12 cycles	78 (±5)
16 layers	Unaged	85 (±3)
	3 cycles	75 (±3)
	6 cycles	65 (±6)
	9 cycles	60 (±4)
	12 cycles	56 (±4)
24 layers	Unaged	69 (±2)
	3 cycles	52 (±5)
	6 cycles	55 (±3)
	9 cycles	40 (±2)
	12 cycles	43 (±4)

14-day wetting/7-day drying process. The stress–strain curve of the unaged and aged GFRP and CFRP specimens after 1, 2 and 4 aging cycles in represented in Figure 10.

As illustrated in Figure 10, in GFRP specimens, the flexural stress–strain curves of unaged and aged specimens are close together and the flexural parameters do not change significantly after cyclic aging. On the contrary, in the CFRP specimens the flexural stress–strain curves are strongly dependent on the number of aging cycles and the slope of the curves decreases with the number of aging cycles. Figure 11 shows variation of flexural modulus of the GFRP and CFRP specimens clearly.

As shown in Figure 11, the flexural modulus of CFRP specimens is more sensitive to the cyclic ageing conditions than the GFRP samples. In the CFRP specimens, the flexural modulus after 4 cycles decrease to 70% of the unaged condition, but in GFRP specimens this parameter decreases only 7% after 4 cycles aging.

In three point bending tests, in addition to the flexural modulus, the flexural strength and the value of flexural strain at failure are also key factors for designing purposes. In the next section, these two parameters are analyzed for different aging conditions.

Flexural strength and maximum flexural strain

Equations (2) and (3) were used to calculate the flexural strength and the flexural strain at failure in three-point bending tests, respectively.

- 7-day wetting/7-day drying process

The variation of flexural strength and failure flexural strain for CFRP specimens before and after cyclic aging are shown in Table 4.

For further analysis of the effect of cyclic aging on the flexural strength and failure flexural strain for the different tested conditions, the normalized flexural strength and the normalized failure flexural strain are plotted as a function of number of aging cycles in Figure 12.

The obtained results show that the flexural strength decreases by increasing the number of aging cycles (see Figure 12(a)). According to Figure 12(a), the effect of cyclic aging on the flexural strength of the 8-layer specimens is less significant comparing with the 16- and 24-layer samples. It is also shown that the variation of the flexural strength for the 16-layer CFRPs is more pronounced than that of the 8-layer specimens and less than that of the 24-layer composite laminates.

Figure 12(b) illustrates the variation of the flexural strain at failure for specimens with different thicknesses. According to the obtained results, the flexural strain at failure increases with the number of aging cycles. From Figure 12(b), it can be found that the variation of the failure

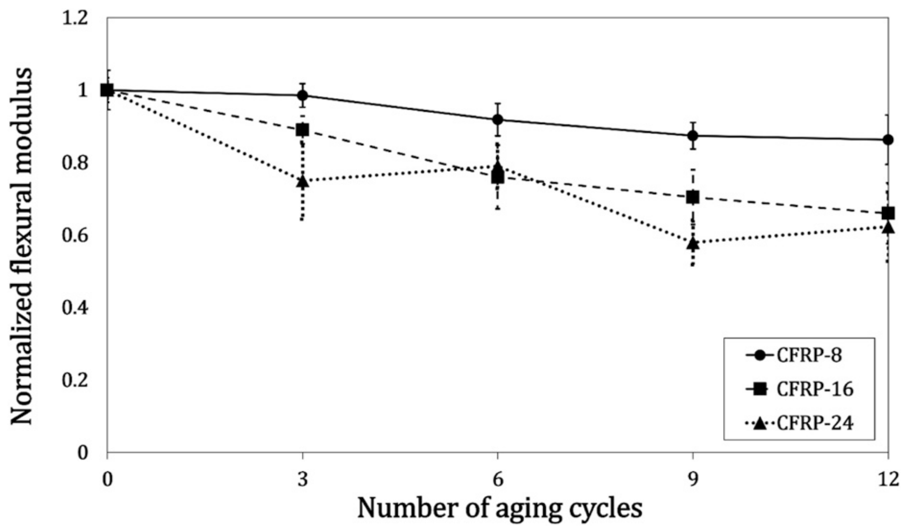


Figure 9. Normalized flexural modulus for different aging cycles and for different specimen thickness of CFRP specimens.

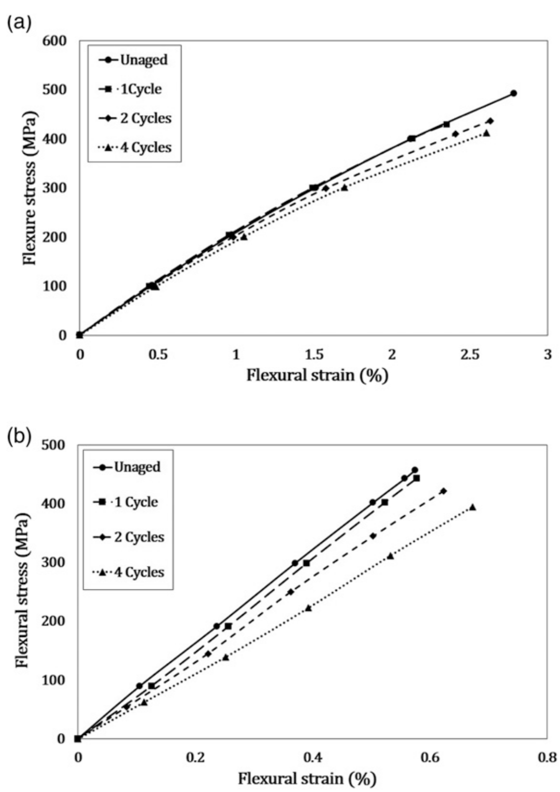


Figure 10. Variation of flexural stress–strain curves for different number of aging cycles for (a) GFRP and (b) 24 layer CFRP specimens.

flexural strain in the 8-layer specimens is less than the other two specimens with 16 and 24 layers. The variation of the flexural properties in different thicknesses shows that the moisture diffusion along the layer interfaces through the lateral sides is more effective than the moisture diffusion through the ply surface which is exposed to the wetting environment. The results show that with the increase of the number of layers in laminated specimens, the effects of moisture uptake on the flexural properties increase. That is the reason why the variation of the flexural properties in the 8-layer specimens is less than that of the other tested CFRPs (see Figure 12). Another influencing parameter, which should be considered, is the effect of shear stresses. It is clear that the amount of shear stresses increases noticeably with the specimen thickness. These stresses in thick specimens should be taken into account in 3-point bending tests. Therefore, in cyclic moisture diffusion conditions where the thickness of the specimens is different, the lateral surface area and consequently the shear stresses are different.

Composite materials in real practice can experience a cyclic ageing environment. Despite the extensive research on single cycle aging of composites, cyclic aging of these materials is less explored.

-14-day wetting/7-day drying process

The flexural strength of the CFRP and GFRP specimens exposed to 14-day wetting/7-day drying cyclic procedure is presented in Figure 13.

As shown in Figure 13, the flexural strength of the CFRP and GFRP specimens decreases by cyclic aging. The influence of cyclic aging on the flexural strength of the CFRP and GFRP specimens are almost similar where for both samples the flexural strength decreases to 85% of the initial values. The obtained results show that this decrease in CFRP specimen is continuous, but for GFRP specimens the

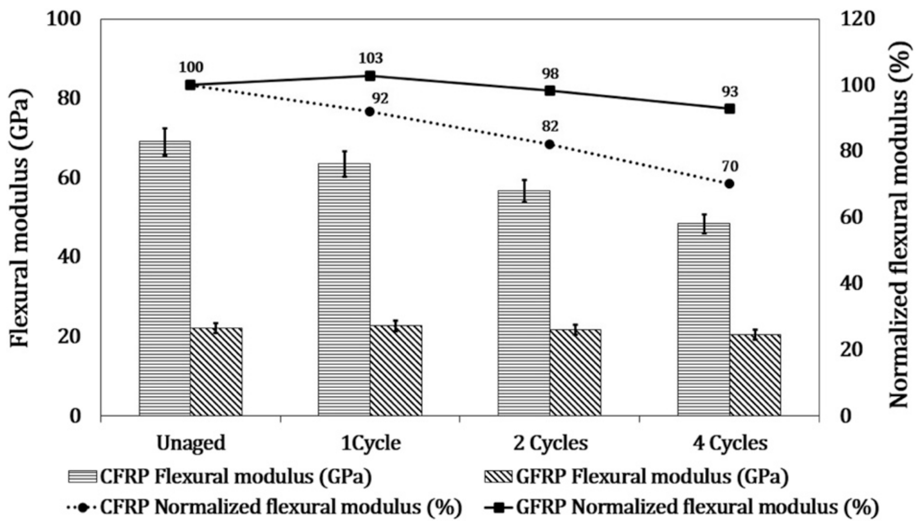


Figure 11. Flexural modulus for different number of aging cycles for GFRP and CFRP specimens.

Table 4. Flexural strength and failure flexural strain for CFRP different specimen thicknesses in unaged and aged conditions.

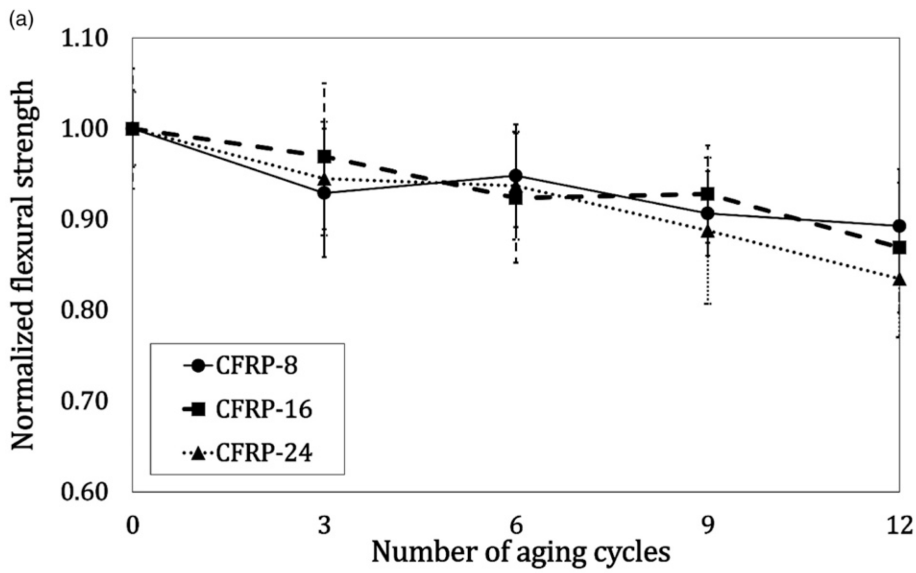
Specimen thickness	Aging conditions	Flexural strength (MPa)	Failure flexural strain (%)
8 layers	Unaged	577 (±24)	0.63 (±0.04)
	3 cycles	536 (±41)	0.64 (±0.04)
	6 cycles	547 (±33)	0.65 (±0.06)
	9 cycles	523 (±27)	0.67 (±0.05)
	12 cycles	515 (±36)	0.67 (±0.08)
16 layers	Unaged	457 (±30)	0.58 (±0.05)
	3 cycles	443 (±37)	0.58 (±0.06)
	6 cycles	422 (±33)	0.62 (±0.06)
	9 cycles	424 (±24)	0.69 (±0.05)
	12 cycles	397 (±31)	0.67 (±0.08)
24 layers	Unaged	507 (±20)	0.71 (±0.04)
	3 cycles	479 (±32)	0.74 (±0.06)
	6 cycles	475 (±33)	0.81 (±0.05)
	9 cycles	450 (±41)	0.84 (±0.07)
	12 cycles	423 (±35)	0.85 (±0.09)

strength decreases sharply after the first aging cycle and then remains almost constant.

As revealed by the previous researchers, the moisture absorption in composite laminates depends on different parameters such as: type of resin,[48] fiber,[48] fiber orientation[49] and laminate thickness.[50] As a result, moisture diffusion coefficient in each composite is a unique parameter that can be investigated using gravimetric tests. Based on the gravimetric test the total moisture content of composite laminate can be measured, but separation of moisture diffusion through the matrix, fiber/matrix interface and interlayer of laminate is difficult and strongly depend of composite parameters such as matrix, fiber and stacking sequence. Previous studies have demonstrated that an increase in laminate thickness leads to an acceleration in the rate of moisture absorption and reduction of flexural properties.[51] This reduction is mainly attributed to the moisture diffusion occurring between the laminates, commonly referred to as interlaminar moisture diffusion.

As claimed by the previous investigators, with increasing of composite thickness the interlayer moisture diffusion and flexural properties reduction increase,[51] simultaneously. Based on obtained experimental results and previous researches[51] in used CFRP specimens, interlayer moisture diffusion is effective and reduce flexural parameters significantly.

It should be noted that in the tested GFRP and CFRP composite laminates, some parameters including the fiber constituents (carbon fiber vs glass fiber), resin chemistry, laminate thickness, and stacking sequence, which affect



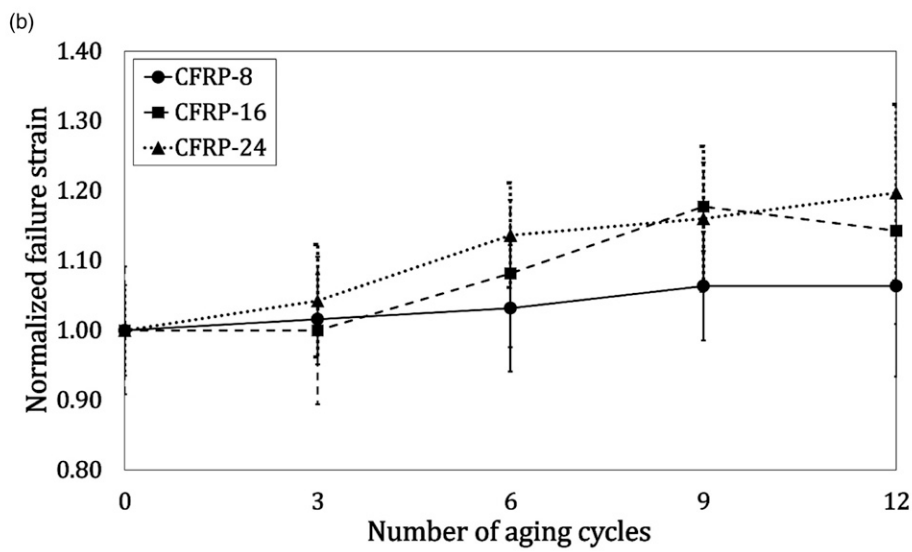


Figure 12. Normalized flexural strength (a) and normalized failure flexural strain (b) for different aging cycles and for different specimen thickness of CFRP specimen.

moisture absorption process are different. Previous researchers revealed that in the same condition moisture absorption of composite with epoxy matrix is more than that with vinyl ester matrix.[52] The results of these investigation showed that the fiber constituents play an important role in moisture absorption process. Researchers claimed that laminates with kenaf fibers absorb moisture more than composites with glass fibers considerably.[52] Another parameters which affects moisture absorption rate in composite materials is interface bonding which is strongly a function of the resin-fiber chemistry.[53] The combination of some fiber-resin chemistry lead to stronger interfacial bonding which results in moisture diffusion reduction.[54] For instance composite laminates including vinyl ester matrix and glass fiber reaches 0.014% moisture uptake after 60 days exposure times which is considerably lower than typical composites.[6] Several researchers have investigated the influence of fiber orientation angle in composite laminates on moisture absorption.[55] In addition, investigations on fiber orientation angle reveal that composites with 0° fiber orientation exhibit significantly larger moisture absorption values compared to those with 90° fiber orientation. Also previous investigations[50] have shown that with increasing the laminate thickness the moisture diffusion rate increases. Therefore, despite the comparable initial stiffness among the two types of composites examined in this study, they exhibited different rates of water absorption and subsequent aging, attributable to the influence of distinct factors on this phenomenon.

Longitudinal strain distribution

To confirm the results obtained in the previous section, the distribution of the longitudinal strain (strain along the length of the tested samples) was experimentally analyzed using the 2D-DIC technique for the 24-layer composite laminates tested in three-point bending conditions. For this purpose, during the bending tests an initial picture of the specimen was correlated with the picture corresponding to an applied load of 200 *N*. With the correlation of the undeformed and deformed pictures, the displacement and strain fields in different directions was obtained. This procedure was performed for the unaged specimen and the aged specimen after 12 aging cycles. During the DIC process in order to capture high resolution reference and deformed images, a macro USM fixed lens and a high resolution camera was used. The resolution of the initial and deformed images was about 2600 × 1700 pixels. The measurement was carried out for the 20 layers which centered in the specimen, as shown in Figure 14 as "Selected domain". After selecting the region of interest, the subset size and the step size parameters for the correlation process were defined 61 × 61 and 9 pixels, respectively. The results are shown in Figure 15.

It can be found from Figure 15 that when the applied load is constant and equal to 200 *N*, the maximum longitudinal strains in the 24-layer specimens before aging and after 12 aging cycles are 3.15e-3 and 5.43e-3, respectively. It means that the maximum longitudinal strain significantly increases after 12 aging cycles and

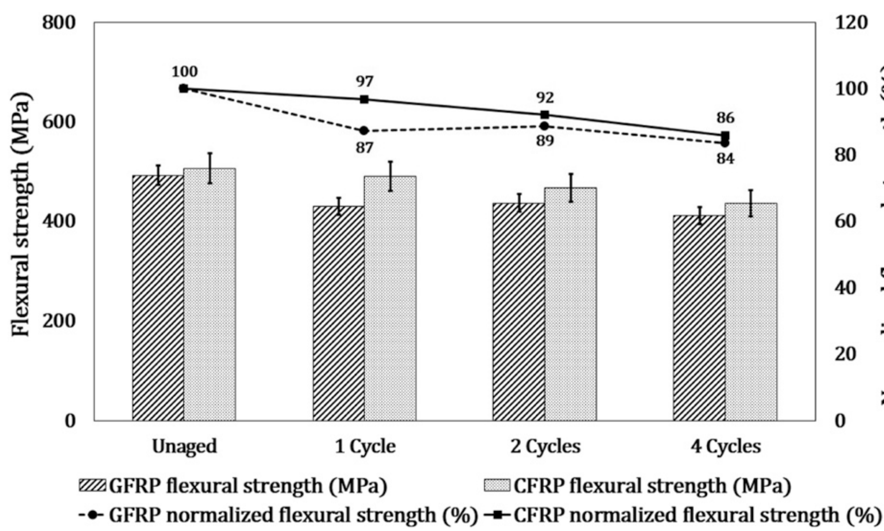


Figure 13. Flexural strength of GFRP and CFRP specimens as a function of the ageing cycles.

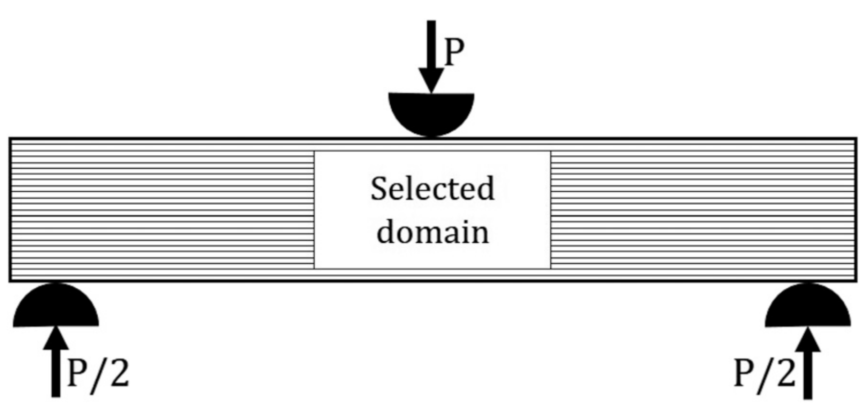


Figure 14. Schematic of region of interest in DIC analysis (dimensions are not scaled).

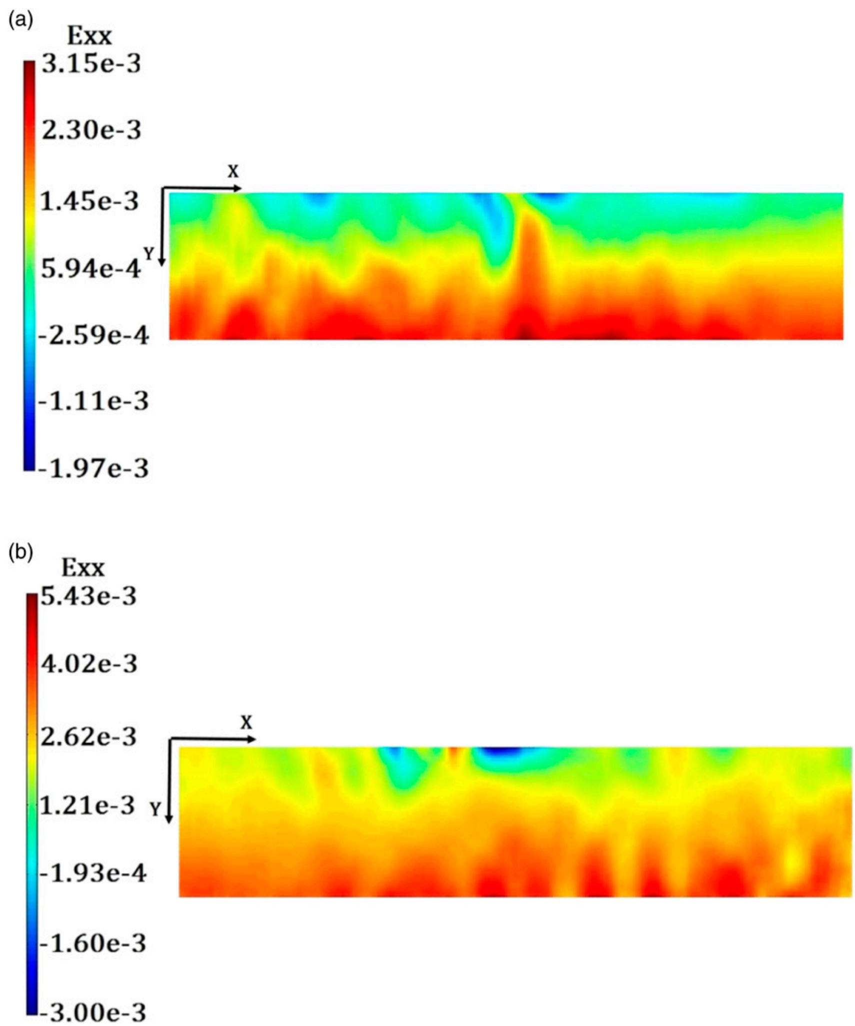


Figure 15. Longitudinal strain distribution of 24-layer specimen for (a) unaged, and (b) after 12 aging cycles in three-point bending tests for an applied load of 200 *N*.

the CFRP becomes more ductile. This conclusion is in agreement with the results already presented in previous sections.

Conclusion

“In this study, CFRP and GFRP specimens sealed on one side were exposed to cyclic aging conditions and tested under tensile and bending loads. The investigation was motivated by the real-life application of composite laminates, which are often covered or sealed on one side by other components, such as adhesive bonding. Based on experimental results variation of tensile and flexural properties of CFRP and GFRP specimens were studied as a function of laminate thickness and aging cycles. Based on the obtained results, the following conclusions were drawn:

(1) For the tested CFRP and GFRP samples with the mentioned stacking sequence, the variation of the elastic modulus for specimens with different thicknesses is negligible in comparison with unaged condition.
(2) It was found that the flexural modulus of CFRP specimens decreases with the number of aging cycles. Flexural modulus of 8-, 16-, and 24-layer specimens reduced by 13%, 34% and 37%, respectively after 12 aging cycles.
(3) The obtained results showed that for the CFRP specimens, the moisture diffusion along the interface of the layers from the lateral sides is more important than the moisture diffusion through the layers from the top surface.
(4) The obtained flexural strength of CFRP laminates for different aging cycles showed that the flexural strength decreases significantly with aging cycles.

(5) The flexural strain at failure increases with the number of aging cycles for CFRPs with different thicknesses. This parameter increases in the 8-, 16- and 24-layer specimens 7%, 19% and 20%, respectively after 12 cycles aging.
(6) For the considered CFRP and GFRP specimens with the same initial flexural stiffness, the flexural modulus and stiffness of CFRP specimens was more influenced by the cyclic aging compared with the GFRP samples.
(7) Although after the first aging cycle a significant reduction was observed in the strength of the GFRP specimens, both the CFRP and GFRP showed the same level of strength reduction after four aging cycles.

Declaration of conflicting interests

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ORCID iDs

M.R. Ayatollahi https://orcid.org/0000-0001-9840-6225
A. Akhavan-Safar https://orcid.org/0000-0002-7168-7079
S. Teixeira de Freitas https://orcid.org/0000-0002-0847-6287

Data availability statement

All data analyzed during this study are included in the manuscript and related datasets are available from the corresponding author.

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